

ANNUAL REPORT

9/11
67925
1N-74-CR
145525
P.20

On: **GROWTH AND CHARACTERIZATION OF BINARY
AND PSEUDO-BINARY III-V COMPOUNDS
EXHIBITING NON-LINEAR OPTICAL BEHAVIOR**

AND

**UNDERGRADUATE RESEARCH OPPORTUNITIES
IN MICROGRAVITY SCIENCE AND TECHNOLOGY**

Contract No.: **NAGW-1563**

Through: **November 30, 1991**

Submitted to: **National Aeronautics and Space Administration
Dr. Brad Carpenter**

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(NASA-CR-192143) GROWTH AND
CHARACTERIZATION OF BINARY AND
PSEUDO-BINARY 3-5 COMPOUNDS
EXHIBITING NON-LINEAR OPTICAL
BEHAVIOR. UNDERGRADUATE RESEARCH
OPPORTUNITIES IN MICROGRAVITY
SCIENCE AND TECHNOLOGY Annual
Report, period ending 30 Nov. 1991
(MIT) 20 p

N93-21363

Unclass

G3/74 0145525

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EXECUTIVE SUMMARY OF PROGRESS REPORT ON:

Growth and Characterization of Binary and Pseudo-Binary Compounds

1. In line with the specified objectives we have developed a Bridgman-type growth configuration in which unavoidable end effects – conventionally leading to growth interface relocation – are compensated by commensurate input-power changes; the growth rate on a microscale is predictable and unaffected by changes in heat transfer conditions.
2. To permit quantitative characterization of the growth furnace cavity (hot-zone) a 3-D thermal field mapping technique, based on the thermal image, is being tested for temperatures up to 1100°C.
3. Computational NIR absorption analysis has been modified to now permit characterization of semi-insulating single crystals.
4. Work on growth and characterization of bismuth-silicate has been initiated. Growth of BSO ($\text{Bi}_{12}\text{SiO}_{20}$) for seed material by the Czochralski technique is currently in progress.

* * * * *

Undergraduate Research:^A Ground based measurements of the wetting behavior (contact angles) of semiconductor melts on substrates consisting of potential confinement materials for solidification experiments in a reduced gravity environment are in progress. Hardware modifications required for execution of the wetting experiments in a KC-135 facility are developed.

GROWTH AND CHARACTERIZATION OF BINARY AND PSEUDO-BINARY III-V COMPOUNDS

August F. Witt

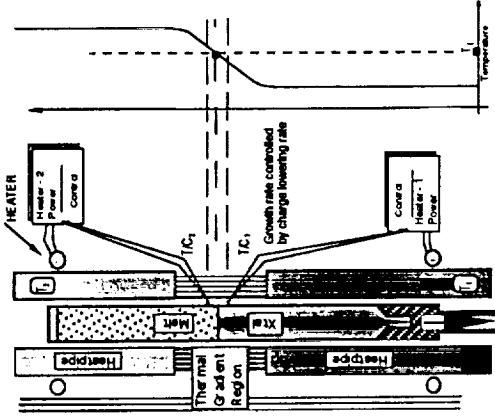
MASSACHUSETTS INSTITUTE OF TECHNOLOGY



Objective: * Identification of gravitational effects on crystal growth, segregation and defect formation as well as determination of their effects on properties which are critical for device applications.
* Development of approaches to the control of critical gravity-related defect formation during growth of compound semiconductor systems.

Approach: * Development of model-based Bridgman growth system with rate control under quantifiable thermal boundary conditions.
* Development of thermal imaging for hot-zone characterization and growth control.
* Development of quantitative near IR transmission microscopy with computational absorption analysis.

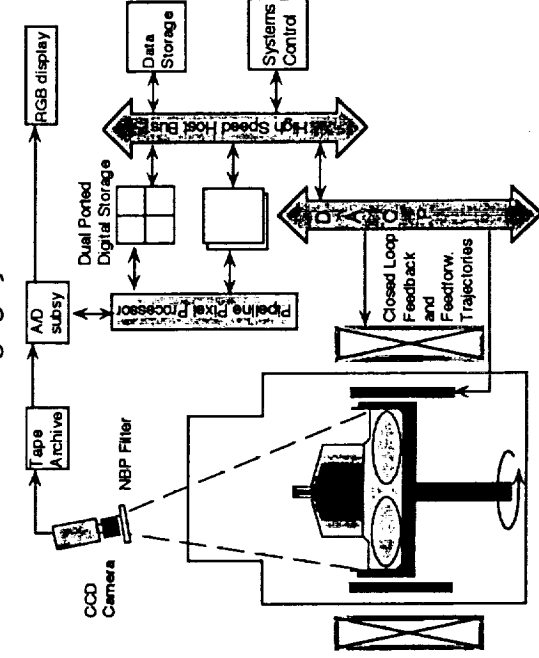
Heatpipe Based Bridgman Geometry with Growth Rate Stabilization



Status:

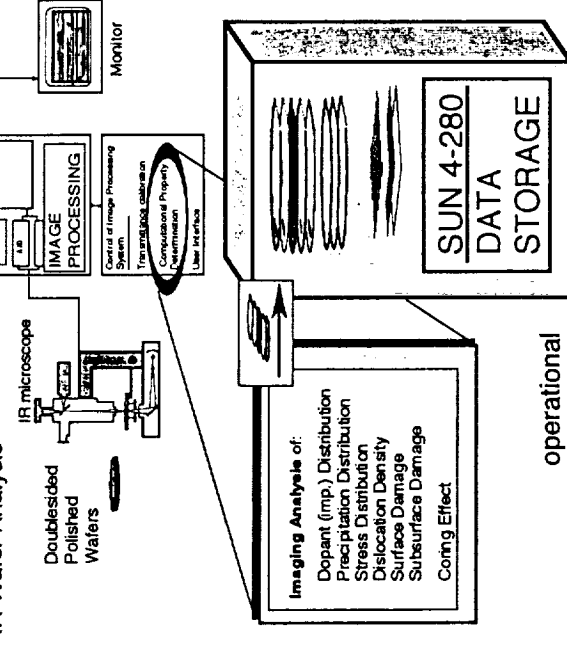
operational

Thermal Imaging System



operational

Computational IR Water Analysis



operational

1. REAL TIME THERMAL IMAGING FOR ANALYSIS AND CONTROL OF CRYSTAL GROWTH

A real time thermal imaging system with temperature resolution better than $\pm 0.5^{\circ}\text{C}$ (1000°C to 1500°C) and spatial resolution of better than 0.5 mm has been developed. It has been applied to the analysis of melt surface thermal field distributions in both Czochralski and liquid encapsulated Czochralski (LEC) growth configurations. The sensor can provide single/multiple point thermal information; a multi-pixel averaging algorithm has been developed which permits localized, low noise sensing and display of optical intensity variations at any location in the hot zone as a function of time. Temperature distributions (gradients) are measured by extraction of data along a user selectable linear pixel array and are simultaneously displayed, as a graphic overlay, on the thermal image.

The thermal imaging technique is currently being modified to permit the characterization of 3D temperature fields in hot zone cavities of Bridgman-type geometry. Thermal imaging, in this modified configuration, will provide quantitative data on the prevailing thermal boundary conditions, their reproducibility and control. Availability of this data is a prerequisite for the conduct of quantitative growth experiments, their theoretical analysis and the interpretation of results; it is considered essential for the characterization of growth hardware to be used in flight experiments, but also important for the characterization of semiconductor processing hardware in a fabline environment.

Introduction and Background

The growth of semiconductor single crystals by the Czochralski pulling technique and, in particular, the growth of compound semiconductors from liquid encapsulated melts, requires knowledge and precise control of the thermal field in the melt and the growing crystal [1,2]. The spatial temperature distribution in the melt is a complex function of the hot zone characteristics, the crystal and the environmental thermal boundary conditions. In conventional growth systems, a single-input-single-output (SISO) closed loop proportional, integral and derivative (PID) controller is used to maintain and/or change a single temperature to accomplish the task of controlling the

temperature distribution in the melt. The sensors widely used for this purpose are thermocouples or single color pyrometers. Their location, by tradition fixed empirically, is well removed from the area of measurement interest: the melt and melt/crystal interface.

Shortcomings of the practiced single point temperature measurements and control relate to their inability of providing critical information on prevailing stability, symmetry and gradients of the thermal fields in the melt about the rotational crystal pulling axis. Thus, conventional temperature sensing can in some instances be used for crystal diameter control but fails to support efforts directed at the minimization of radial and micro-segregation as well as the suppression of stress related defect formation.

Theoretical Basis for Image Interpretation, Hardware Configuration and System Architecture

The relationship between changes in optical intensity (I), temperature (T) and wavelength (λ) is established by Wien's approximation [eq. 1] to Planck's radiation law.

$$I(\lambda, T) = C_1 \frac{\epsilon(\lambda, T)}{\lambda^5} e^{\frac{-C_2}{\lambda T}} \quad [\text{eq.1}]$$

C_1 and C_2 are constants, 3.7403×10^{-4} watt μm^2 and 1.438×10^4 $\mu\text{m}^\circ\text{K}$ respectively and ϵ is the emissivity of the melt. The operation of the thermal imager is based on the theoretical principles of single color optical pyrometry. The high temperature scene is viewed in near normal incidence by a high resolution (512 x 512 pixels) low noise (–60 dB) charge coupled device (CCD) camera (RCA, TC2855). A narrow bandpass filter (3 nm FWHM, centered at 633 nm) is placed between the scene and the camera. In this way, an array of 1/4 million discrete optical pyrometers are available to characterize the scene with a spatial resolution of better than 0.5 mm. The transmission wavelength of the filter was chosen to approach the peak in the spectral sensitivity of the CCD element

while maintaining a steep slope on the I vs T (gray body) curve of the emitting (melt) surface.

A schematic of the thermal imaging architecture and a preliminary control structure (fig. 1) includes a growth system, a CCD camera and narrow bandpass filter, an image processing subsystem with high resolution analog RGB monitor and a host computer with integral high speed data acquisition and control capability (Masscomp MC-5510).

The camera generates information at 30 frames/second using a standard RS-170 (B/W) configuration. The analog video information is archived on 3/4" video tape (Sony BVU-800) and sent to the analog to digital subsystem where gain and dc offset are adjusted to permit operation at high gain for maximum sensitivity (temperature resolution). The signal can be pre-processed with user definable look up tables (LUT) to correct for inherent nonlinearities within the camera and is finally digitized to 8 bits (256 gray levels). Image processing functions are carried out in the pipelined pixel processor. These include calculation of two dimensional convolutions and temporal averaging with gain adjustment. Digital storage units (image memory) are used to hold starting, intermediate and final images during these procedures. Image transfer to and from the storage locations is provided by the pipelined pixel processor under software control. A unique capability of the imaging system is the dual ported nature of the digital storage units. The image memory thus appears as extended system memory to the host computer. As a result, image data can be accessed simultaneously by the pipelined pixel processor and the host CPU. In this way, parallel processing of the information can be accomplished with no performance penalty; the data extracted from the images can be processed while image processing functions continue. Real time graphics overlays of extracted and filtered image data and linear convolutions are produced in this manner. Two of the digital storage units are configured as a 16 bit pair to hold high precision intermediate results produced during certain image processing calculations (e.g. 2-d convolutions and temporal averaging). A third 1024 x 1024 x 8 image memory unit is configured as four contiguous 512 x 512 x 8 digital storage units

which act as entrance and exit points for image processing calculations. Provision is also made for storage of select individual digital images on magnetic disk.

The processed images are sent back (in real time) through the A/D subsystem where a second LUT is used to false color the outgoing monochrome signal; the human eye is more sensitive to small changes in color than equivalent changes in brightness (gray level). The LUT is defined to map low intensities to blue and high intensities to red with a linear interpolation of the complete 8 bit dynamic range. In this way, a complete spectrum between blue and red corresponds to intensities from 0 to 255 gray levels. The output is displayed (with the graphics overlays) on a high resolution RGB monitor.

Optical intensity information is selectively extracted from the thermal image, processed by the host CPU, displayed as a graphic overlay on the real time imaging signal and then passed, via the host, to the Data Acquisition and Control Processor (DACP). For temperature control purposes, this data is scaled and offset to conform to the output characteristics of a platinum/platinum-10% rhodium thermocouple and sent from a digital to analog (D/A) converter to the analog temperature control system on the console.

Thermal Imaging of High Temperature Semiconductor Melts

For thermal imaging geometry in Czochralski configuration the CCD camera by necessity is mounted off-axis and non-normal to the melt surface. As a result, the contribution of low level internal reflections from the melt surface to the image is minor. The high temperature scene comprises the melt, crucible/susceptor, heater elements and crystal. The portion of the scene containing optical information convertible to temperature is provided by the flat surface of the melt.

A raw (unprocessed single frame) thermal image of a conventional liquid encapsulated melt prior to seeding depicts high melt temperatures as bright and lower temperatures as progressively darker. Temperature data can be extracted from this scene according to user specification, can be processed and displayed in real time as a

radial temperature profile, for example. The image can also be processed by a linear convolution to remove unavoidable high spatial frequency components; the data processing is off loaded from the pipelined pixel processor to the main CPU so as to maintain real time image processing performance.

Isotherms, corresponding to constant intensity contours, are revealed by highlighting selected gray levels. Their diffuse appearance is attributable to high spatial frequency noise which can be eliminated on a full frame basis. For this purpose, a two dimensional convolution is performed in n frame times where n is the the number of non-zero elements in the convolution kernel (typically 8 of 9 in a 3×3 matrix). Thus, a complete convolution is accomplished in 8 frame times or $8/30$ second. The convolved image does not exhibit high spatial frequency noise; isotherms appear correspondingly in a coherent form.

The foregoing thermal imaging capabilities are characterized by a view of the scene and display of the data at a particular point in time. Another approach, found to be useful for systems control purposes, is to extract and display intensity (temperature) data from an $n \times m$ array of pixels as a continuous function of time.

System Calibration

Two techniques were employed to calibrate the thermal imager. Both permit the relative determination of temperature; i.e. temperature changes/differences are measurable quantitatively. In the first approach, a magnetically stabilized melt was held at constant temperature using a conventional temperature control scheme where the sensing thermocouple was in close proximity to the heater. Three gray levels (isotherms) were highlighted at 20 gray level intervals. The choice of these absolute intensities was made so as to indicate the optical emission at three positions on the melt surface: one near the melt periphery, one at approximately the half radius position and one near the center of the melt. The temperature was subsequently increased, forcing these highlighted gray levels to move toward the center until, at temperature $T + 10^\circ \text{C}$, the innermost (coldest) isotherm has all but disappeared and its previous position (at T) had been taken by the second (middle) isotherm. This superposition of

isotherms was repeated once more as the temperature was again ramped. At $T + 20^{\circ}\text{C}$ the third isotherm (originally near the crucible wall) was located in the same position as that of the first isotherm at temperature, T . Accordingly, over this temperature range, 20 gray levels correspond to a difference of 10°C ; i.e., the sensitivity of the thermal imager is given as 0.5°C per gray level. The second calibration was conducted by imaging the bottom of the graphite crucible susceptor with a thermocouple to measure the imposed temperature changes at the half radius position. Using the algorithm described above for measuring the optical intensity at a point, the gray level output of the thermal imager (averaged 9×9 pixel array) was amplified, scaled, processed by a digital to analog converter and directly compared to the response of the thermocouple. The relationship between the outputs of the thermocouple in $^{\circ}\text{C}$ and the thermal imager in volts indicates 20 gray levels for every 10°C .

Temperature Control by Thermal Imaging

Conventional temperature control schemes in growth configuration are based on sensor locations well removed from the critical area of interest. Using the thermal imager as a single point sensor the temperature of the growth system was controlled from within the crucible and, thus, from a more process sensitive position. The intensity (temperature) information from the imager was re-scaled (gain and dc offset) so that the output of the D/A converter, after processing by a 1000:1 voltage divider, corresponded directly to that of a Pt/Pt-10%Rh thermocouple. In this way, the signal could be fed to the conventional, analog PID controller which had previously been used to control a thermocouple placed near a heater element. The output of the thermocouple located within the crucible and the old control thermocouple (at the heater) were recorded. In both cases, the change in output is found to correspond to the temperature changes made at the controller indicating that the signal from the thermal imager successfully tracked and measured the temperature response of the system.

Discussion

One of the outstanding features of thermal imaging for high temperature crystal growth is in the great facility with which time dependent changes in temperature can be recorded and, in various ways, used as input for systems control. The approach is non-invasive and applicable to point, line and area measurements through software selection.

The interpretation of the output of the thermal imager as temperature information is more complicated. It is based on three fundamental assumptions. First, the high temperature scene is viewed in normal incidence to eliminate effects of internal reflections. Second, the emissivity of the melt does not change over the temperature range of interest. And, third, the characteristics of the optical path are time independent. In this context, it is of interest to analyze the degree to which these assumptions are met in the present experimental configuration and what effect any deviation from these assumptions have on image interpretation.

In the reported configuration (fig. 1) the camera views the melt at an inclination of about 5° to the normal. The effect of this non-normal incidence is evident during observation of metallic (specular) melts where isotherms appear shifted from a centro-symmetric position. No such effect is observed, however, when imaging an empty graphite crucible (non-specular); isotherms appear concentric about the center of the crucible. This finding suggests that, when imaging the melt surface, high temperature information from the crucible wall is being reflected by the specular melt to the thermal imager. Work is in progress to negate this effect by subtraction of a reference image containing information characteristic of the reflection.

There is no indication that the emissivity of the melt changes measurably over the temperature range encountered during a typical growth experiment. However, a change of the 'effective' emissivity of the melt does occur as a result of changing optical effects associated with gas bubbles in the encapsulating layer. Compensation of this effect, for example during crystal diameter and/or temperature sensing, is being approached by

modifying the temporal averaging algorithms for inclusion in the systems control software.

The characteristics of the optical path are subject to a significant aberration from assumed behavior over the course of a growth experiment. During this time: (a) the transmission through the window used to view the melt is subject to time dependent change due to deposition of volatile constituents in the system. (b) the optical transparency of the encapsulant changes as its chemistry is modified by the interaction of the melt with its environment. These effects can be corrected for by image subtraction if the changes to the characteristics of the optical path are reproducible and well characterized. It is preferable, however, to approach this problem through elimination of the causes rather than the effects.

References

1. K. Morizane, A.F. Witt and H.C. Gatos, J. Electrochem. Soc. 114 (7) 738 (1967).
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Figure Caption

1. Thermal imaging architecture and preliminary control structure for LEC growth of GaAs. The use of closed loop feedback and feedforward trajectories are discussed in detail in reference 4. Non-traditional control elements include the heat exchange system (HES) around the growing crystal and the superconducting magnet around the furnace.

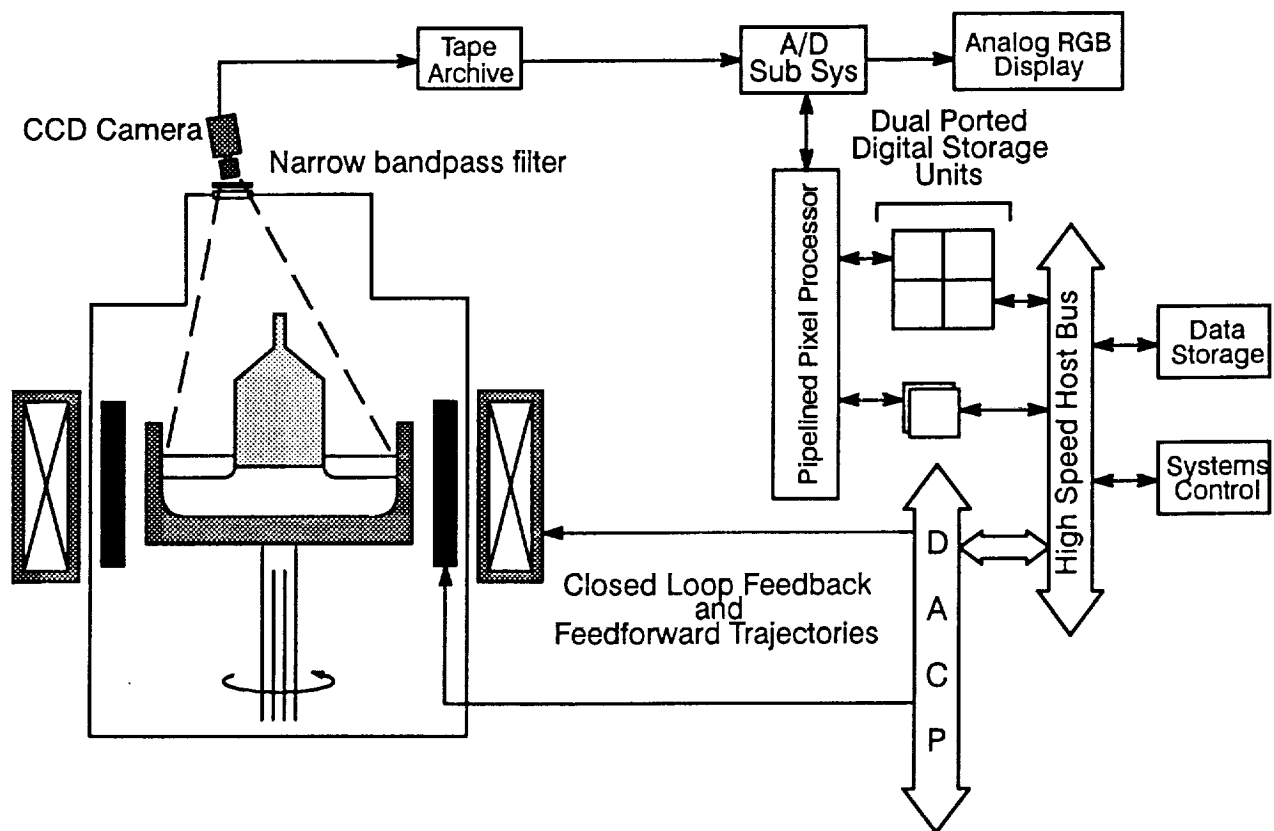


Figure 1

2. OPTICAL APPROACH TO THE QUANTITATIVE CHARACTERIZATION OF CRYSTAL, SEGREGATION AND DEFECT FORMATION

The meaningful exploration of the potential of reduced gravity environment for the advancement of crystal growth is complex. It is to a significant extent contingent on the conduct of reproducible experiments in a quantifiable environment. However, most of all it depends on our ability to extract from grown matrices quantitative analytical information on a scale commensurate with that of gravitation related segregation effects and defect structures.

A new optical approach, based on NIR microscopy supported by computational image analysis and contrast enhancement, has been developed and applied to the characterization of elemental and compound semiconductors. This approach permitted, for the first time, a quantitative microsegregation analysis of GaAs and InP; using NIR dark field illumination in transmission mode makes it now possible to detect sub-micron precipitates in semi-insulating GaAs. The developed techniques, providing for rapid, quantitative, non-destructive analysis, have been shown to be fully compatible with telescience operation. It is currently being adapted to characterization of non-linear optical materials in the oxide family.

The rapid advance of silicon based technology compared to that of compound semiconductors reflects the relative simplicity of this system. III-V and, even more so, II-VI compound semiconductors, while exhibiting outstanding optical and electrical properties, are also subject to complex defect formation¹ which affects adversely performance and yield of devices. Progress in compound device technology for this reason has so far been slow. Difficulties arise primarily from the lack of techniques for identifying quantitatively and in a non-destructive manner critical chemical and crystalline defects which prevents the subsequent establishment, through appropriate correlation analyses, of the property requirements for device fabrication. These analytical complications are also directly responsible for our inability to establish crystal growth control objectives directed at minimizing critical defect formation through application of appropriate growth conditions.

The non-availability of analytical techniques for quantitative, sensitive, high resolution defect analysis has also impeded the meaningful study of gravitational effects

on crystal growth and segregation. While it now appears well established that gravitational effects are directly and/or indirectly responsible for the formation of the vast majority of critical defects during crystal growth, the exploration of the potential of reduced gravity environment in efforts directed at clarifying related cause and effect relationships has been conspicuously slow. Results from crystal growth experiments conducted in space have been of questionable impact primarily because of our inability to identify unambiguously on a micron and submicron scale gravitational effects on crystal growth and segregation^{2,3}.

The present work, development of a non-invasive quantitative semiconductor characterization technique⁴ with spatial resolution in the micron-range, was undertaken to permit the establishment of application dictated property requirements for device fabrication and to advance our exploration of the potential of reduced gravity environment of materials research.

Experimental

The analytical technique developed for rapid, non-destructive defect analysis in semiconductors is based on quantitative image analysis in conjunction with near infrared (NIR) transmission microscopy. NIR microscopy provides an image of the semiconductor sample (0.5–2 mm thick) reflecting any local variations in the absorption coefficient across the field due to the presence of defects. Qualitatively this image provides an excellent representation of material uniformity. It contains in addition, however, information which can be used for the quantitative determination of local free charge carrier concentrations and lattice stress conditions, for example.

NIR microscopy relies on an imaging device such as a CCD camera or a silicon vidicon camera to detect transmitted radiation. To quantify the image, the output of the camera is used as input to a digital image processor where the signal is digitized into a 512 by 480 pixel array with a dynamic range of 256 gray levels. The image processing system provides for multiple image storage and near real time whole image mathematics capability. The approach taken for data calibration is to increase the gain of the system so that the entire range is distributed over the limited transmittance range

exhibited by the sample; using neutral density filters, the measured gray level value can then be equated to transmittance. Comprehensive defect mapping can be accomplished by determination of the IR (and, as applicable, the visible) spectrum for a given material by means of an FTIR spectrometer and identification of chemical defects, for example, through their characteristic absorption peaks (fig. 1). The spatial distribution of these defects is subsequently analyzed and the corresponding data are stored in digitized form with their spatial coordinates. The fundamental characteristics of computational optical defect analysis are presented in fig. 2. The mode is non-invasive, applicable to both the micro and macro scale with a maximum spatial resolution approaching 1 μm ; data storage with spatial coordinates is provided for with complete image analysis requiring a fraction of one second.

The developed optical technique has so far been applied to dopant concentration analysis, stress analysis, dislocation density measurements in conducting and semi-insulating matrices, surface damage measurements and precipitation analyses. Work is in progress on the determination of lattice damage associated with ion implantation and on the identification of defect propagation into epitaxial layers.

The non-destructive nature of the computational image analysis in combination with digital data storage provides a means for the conduct of a statistical correlation analysis between device characteristics and spatially coincident wafer characteristics. Such analyses can be accomplished upon subjecting the analyzed wafer to device processing and mapping the intermediate processing steps as well as the spatial distribution of yield and performance of devices.

Results

An NIR transmission micrograph of commercial Te-doped GaAs reveals that microsegregation effects associated with LP-LEC growth result in compositional fluctuations which, over microdimensions, approach and in some instances exceed one order of magnitude. This finding is contrasted by the general belief that such fluctuations are in the range of ± 15 to 30% of the average doping value. It is also found that, contrary to expectation, the striation pattern is discontinuous along the crystal

diameter. On the basis of this quantitative segregation analysis, it is concluded that growth is subjected to turbulent free density driven melt convection; this results in localized thermal perturbations which penetrate the solute boundary layer and lead to melt-back. Subsequent regrowth and dopant incorporation appear largely controlled by fluid dynamics at the phase boundary and cannot be interpreted on the basis of the Burton, Prim and Slichter theory. The visibility of the complex dislocation network in NIT transmission microscopy is attributed to decoration of the dislocations by dopant elements. It should be pointed out that a comparative analysis of dislocations in GaAs grown by the LEC technique with GaAs grown by the horizontal Bridgman technique revealed fundamental differences in density, distribution and geometry. Subjecting the wafer to a mild dislocation etching solution it was possible to ascertain the correspondence of etch pits with dislocation terminations.

Analysis of semi-insulating GaAs indicated transparency to 1 μm radiation in bright light transmission mode. Using dark field NIR illumination, extensive scattering centers of submicron dimensions are observed. The scattering centers, precipitates, appear as decorations of dislocations and thus delineate the location of these otherwise invisible defects. Quantitative IR absorption analysis (AIAA) is presently applied to the defect characterization of bismuth-silicate ($\text{BSO} - \text{Bi}_6\text{SiO}_{20}$), a compound considered for melt growth in a reduced gravity environment. The large band gap provides for absorption in the visible spectral regime as well as in the IR. Detailed characterization is in progress.

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3. DEVELOPMENT OF HEAT PIPE BASED BRIDGMAN GROWTH SYSTEM

Hot Zone Characterization by Thermal Imaging

The construction and operation of a Bridgman-type growth system, based on computational heat transfer analysis, requires our ability to characterize quantitatively the 3-dimensional thermal field of the furnace cavity under operating conditions. The use of thermocouple arrays, as currently practiced, is considered as inadequate and necessitated the development of an alternate approach: thermal imaging.

Thermal imaging, developed in this laboratory for temperature measurement and growth control of LEC systems, is currently being modified to permit hot zone characterization. The approach taken involved IR imaging of the radiation emitted from the upper surface of a graphite rod being lowered through the cavity of a heat pipe based Bridgman furnace. Thermal data are being stored, converted and depicted as axial temperature profiles of the cavity. The available database for the materials involve3d is expected to permit the quantitative determination of thermal boundary conditions in the growth cavity. The analytical approach taken is also expected to yield data on heat transfer characteristics of the growth system.

4. COMPARATIVE ANALYSIS OF THE WETTING BEHAVIOR OF UNDOPED AND DOPED SEMICONDUCTOR MELTS ON THE GROUND AND IN A REDUCED GRAVITY ENVIRONMENT

Roy Rasera and Sherry Ipri

Undergraduate Research Opportunity in Microgravity Crystal Growth

The experiment is being designed to observe the wetting behavior of semiconductor melts through the measurement of the contact angle between molten samples and their confinement material (fig. 1). These angles and their respective environmental parameters will be added to a microgravity crystal growth data base. The data base will aid researchers in designing procedures to optimize growth experiments on semiconductors under microgravity conditions.

Experimental

A horizontally oriented transparent gold furnace is currently used as a transparent high temperature environment. A light source in back of the furnace provides background illumination for photography. In this configuration the light outlines the test sample within the furnace and the contact angle between the sample and its confining material becomes measurable. The camera is directly connected to a video monitor and a 3/4-inch u-matic VCR for observation and recording, respectively. Experiments involving InSb, GaSb, GaInSb and Ge are in progress. The system is being tested using "flattened ball bearings" on reflecting surfaces (confinement materials).

For experiments on the KC-135 some changes will most likely have to be made. First, the furnace will be oriented vertically to reduce gravitational stresses on the furnace and the sample. For safety reasons, the furnace will need to be modified. These modifications must allow for videotaping of the sample, as well as attachment of an illumination source. The camera must remain accessible to allow for manual adjustments; it must also remain connected to the video screen/VCR so that the picture quality can be adjusted and the wetting behavior recorded. Some additions to the setup will have to be made. A manual system for insertion and retrieval of the samples is under construction. A method of safe storage for the samples is still to be developed.

Related Publications

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